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SEMI-SOLID PROCESSING OF CHOCOLATE AND COCOA BUTTER
Modelling Rheology and Microstructure Changes During Extrusion

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This paper describes the development and application of a model that is capable of describing the extrusion behaviour of semi-solid chocolate. The model is based on the flow of a perfect plastic material within an extrusion die. During the isothermal extrusion process, work is carried out and a model has been developed to correlate the work done with the transformation of part of the crystalline triglyceride molecules to the liquid state. Numerical results from finite element calculations are presented for the variation of liquid fat content as a function of position within the extrusion die for a single extrusion, and analytical results are presented for the variation of the average liquid fat content and of the extrusion pressure for sequences of extrusions. The model demonstrates that, in certain situations, changes of a microstructural variable, rather than temperature changes, are necessary in order to describe the dissipation of work during a forming process.

Keywords: chocolate; cocoa butter; forming; extrusion; soft solid; modelling; plasticity; phase transition.

INTRODUCTION

In the companion paper to this one (Engmann and Mackley, 2005), background and experiments were described in relation to the isothermal 'cold extrusion' of chocolate and cocoa butter. The paper presented experimental results that gave insight into the mechanism associated with this unusual process where chocolate can be shape formed in the semi-solid state. A key finding of the paper was that the extrusion history of the chocolate influenced the extrusion pressure and that this was correlated to the amount of liquid fat present in the chocolate. In this paper we develop a model for the extrusion of semi-solid chocolate where the amount of work done influences the liquid volume fraction of fat present.

We consider the extrusion of semi-solid chocolate through an axisymmetric die with a geometry shown schematically in Figure 1. Previously, both slit and axisymmetric cold extrusion of chocolate has been modelled using a single-parameter perfect plastic model having a plastic yield stress τ_y and this greatly simplified comparison between model predictions and experimental data for flows occurring in extrusion dies (Mulji and Mackley, 2003, 2004). Analyses of this type are well established in metal forming science (see e.g., Avitzur, 1968; Chakrabarty, 1987; Kobayashi *et al.*, 1989; Wagoner and Chenot, 2001) and have recently been applied to ceramic pastes and other soft solids (Horrobin and Nedderman, 1997; Horrobin, 1999).

It has been reported previously (Crook, 1997; Ovaici, 1999) that the yield stress of semi-solid chocolate depends on extrusion temperature and composition of the chocolate, and some typical data on the way the yield stress depends on temperature taken from this work is replotted here in Figure 2.

It is also well known that the state of the fat matrix in chocolate (consisting mainly of triglyceride molecules) depends strongly on temperature (Timms, 1984; Beckett, 1999, 2000; Walstra, 2002). As the temperature increases, more of the fat matrix becomes liquid and data on the change of liquid fat component of two milk chocolates and one cocoa butter as a function of temperature are shown in Figure 3. The increase in the liquid fat content, $\phi(\equiv m_{\mathrm{fat,liquid}}/m_{\mathrm{fat,total}})$ with increasing temperature corresponds directly to the decrease in crystalline component of the fat matrix and is the primary cause for the progressive softening of chocolate from a brittle solid at 0°C to a viscous liquid at 35°C.

Of particular relevance to the model developed in this paper is the experimental observation that the cold extrusion process is essentially isothermal (Beckett *et al.*, 1994;

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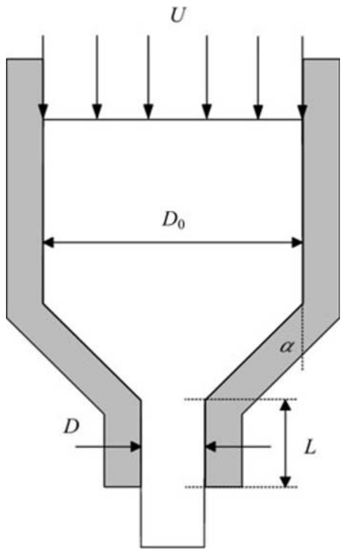

Figure 1. Axisymmetric extrusion geometry.

Crook, 1997) and that the liquid fat phase component of the chocolate, or cocoa butter extrudate increases as a consequence of extrusion (Mulji *et al.*, 2003; Engmann and Mackley, 2005). Our model assumes that the liquid fat content ϕ is the key microstructural variable during cold extrusion of chocolate and that the dissipation of work during extrusion processing is predominantly linked to a temporary phase change of parts of the crystalline fat to the liquid state, which in turn modifies the yield stress of the material.

MODEL

Rheological Constitutive Equation

The rheological behaviour of the chocolate is described by a perfect plastic constitutive equation. Deformation of such a material takes place if,

$$\frac{1}{2}\sum_{i=1}^{3}\sum_{j=1}^{3}\tau_{ij}\tau_{ji} = \tau_y^2 \tag{1}$$

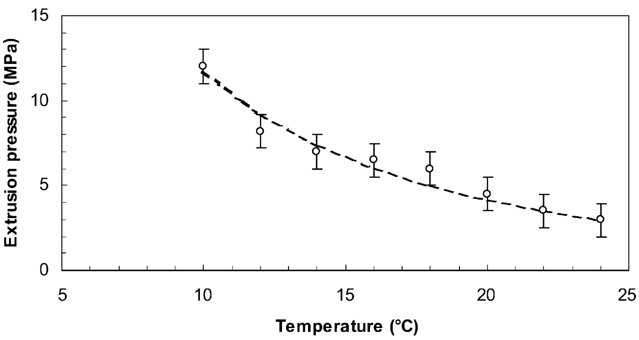

Figure 2. Extrusion pressure as a function of initial temperature for a particular extrusion die geometry and flowrate ($D_0/D = 15$ mm/4 mm, $L/D = 4$ mm/4 mm, $\alpha = 45°$, $Q = 5 \times 10^{-7}$ m^3 s^{-1}). Data replotted from Ovaici (1999).

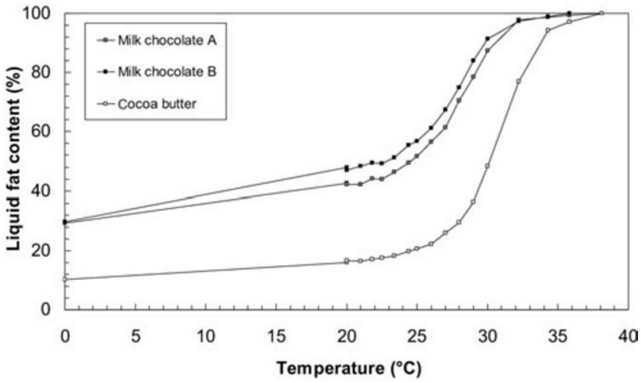

Figure 3. Dependence of liquid fat content with temperature, for two different milk chocolate compositions and a cocoa butter, as measured by pulsed nuclear magnetic resonance (p-NMR).

where τ_{ij} are the components of the deviatoric stress tensor ($\tau_{ij} = \sigma_{ij} + p$) and τ_y is the plastic yield stress. In regions of the material where the LHS of (1) is less than τ_y^2, only rigid-body motion takes place, but no deformation. This is called the 'von Mises criterion' (see e.g., Wagoner and Chenot, 2001). The deviatoric stress (τ_{ij}) and rate of deformation (D_{ij}) components are related by the equation

$$\tau_{ij} = \tau_y \frac{D_{ij}}{\sqrt{(1/2)\sum_{i=1}^{3}\sum_{j=1}^{3} D_{ij}D_{ij}}} \tag{2}$$

from which it can be shown that the stresses τ_{ij} are independent of the rate of deformation as long as the ratios between the components D_{ij} are constant. Elastic deformation is assumed to be negligible in this perfect plastic model, which is a good assumption for chocolate since its elastic shear modulus is much lower than the yield stresses calculated from extrusion pressures. Equations (1) and (2), together with the equations expressing conservation of mass for incompressible materials ($\sum_{i=1}^{3} D_{ii} = 0$) and conservation of momentum in absence of inertial and body forces ($\sum_{j=1}^{3} \partial\sigma_{ij}/\partial x_j = 0$) form the system of governing equations for the mechanical problem to be solved.

Contact Condition at the Material-Die Interface

In the model developed in this paper, shear stresses at the interface between the chocolate and the extrusion die are assumed to be negligible. Effects of constant and of velocity-dependent friction coefficients have been considered in a separate work (Engmann, 2002) and for chocolate flowing through steel, aluminium or brass extrusion dies, the wall shear stresses at temperatures near ambient have been found to be at least one order of magnitude less than τ_y.

Dependence of Plastic Yield Stress on Liquid Fat Content

The yield stress of a highly concentrated solid–liquid suspension such as chocolate depends on the volume fractions of the liquid and solid phases. We use the liquid fat content ϕ ($= m_{fat,liquid}/m_{fat,total}$) as the key microstructural

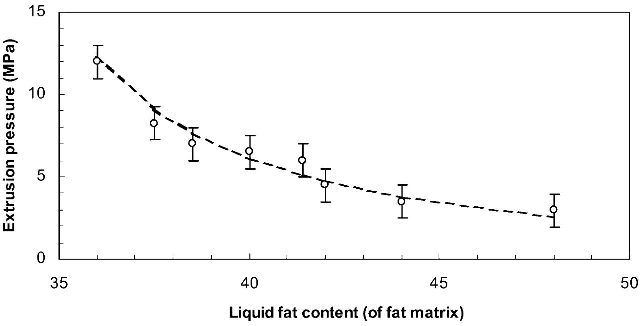

Figure 4. Extrusion pressure as a function of initial liquid fat content for a particular extrusion die geometry ($D_0/D =$ 15 mm : 4 mm, $L/D =$ 4 mm: 4 mm, $\alpha = 45°$). Extrusion pressure data from Ovaici (1999).

variable governing the rheological behaviour. The exact relationship $\tau_y(\phi)$ is unknown *a priori* (and difficult to measure directly). For guidance, we can use the temperature dependence of the extrusion flow pressure for a particular extrusion geometry (Figure 2). Combining this with the liquid fat content-temperature data in Figure 3 allows us to replot the extrusion pressure in terms of liquid fat content, as shown in Figure 4.

Change of Liquid Fat Content as a Result of Work Dissipation

The rate of work per unit volume done locally by the internal stresses during plastic deformation can be calculated from the stress ($\boldsymbol{\sigma}$) and rate-of-deformation ($\boldsymbol{D}$) tensors as

$$\dot{w} = \boldsymbol{\sigma}{:}\boldsymbol{D} = \sum_{i=1}^{3}\sum_{j=1}^{3}\sigma_{ij}D_{ij} \tag{3}$$

This work done increases the local internal energy of the material (changes of the kinetic energy can be neglected), and one may then consider a number of different effects (see Figure 5) of this energy increase:

(a) A *temperature increase with no phase transition* of crystalline triglyceride molecules from the solid to the liquid state,

$$\dot{T} = \frac{\dot{w}}{\rho \cdot c} \tag{4}$$

where ρ is the density of the chocolate or cocoa butter, c its specific heat capacity at the current temperature and liquid fat content, and $\dot{T}$ the rate of temperature increase. As will be shown below, this equation predicts a significant temperature increase after an extrusion that should be easily measurable. This is contradicted by the experimental observations.

(b) An *isothermal phase transition* of crystalline triglyceride molecules to the liquid state,

$$\dot{w} = \rho \cdot w_f \cdot H_m \cdot \dot{\phi} \tag{5}$$

where H_m is the average specific enthalpy of phase transition from the crystalline to the liquid state for triglyceride molecules, w_f the mass fraction of triglycerides in the chocolate, and $\dot{\phi}$ the rate of increase of the liquid fat content.

(c) A *temperature increase* with a *concurrent phase transition* of crystalline triglycerides to the liquid state

$$\dot{w} = \rho \cdot \lfloor c \cdot \dot{T} + w_f H_m \dot{\phi}(\dot{T}) \rfloor \tag{6}$$

This equation requires a relationship for the rate of phase transition $\dot{\phi}$ as a function of the rate of temperature change $\dot{T}$, where (a) and (b) are special cases with $\dot{\phi} \to 0$ and $\dot{\phi} \to \infty$, respectively. If we assume that the process is *close to equilibrium*, then the $\phi(T)$ relationship observed for slow melting (e.g., from p-NMR data) can be used to estimate $\dot{\phi}(\dot{T})$. Most likely, however, the process is far from equilibrium and the relationship not known *a priori*.

An isothermal phase transition (model b) represents the best approximation to the existing data, since no significant temperature changes have been recorded directly after extrusion, while significant changes of solid and liquid fat contents have been documented (Engmann and Mackley, 2000; Mulji *et al.*, 2003). The gradual temperature rise *after extrusion* and the concurrent re-crystallization of triglyceride molecules are also qualitatively explained by this model. Consider Figure 6 that shows the isothermal

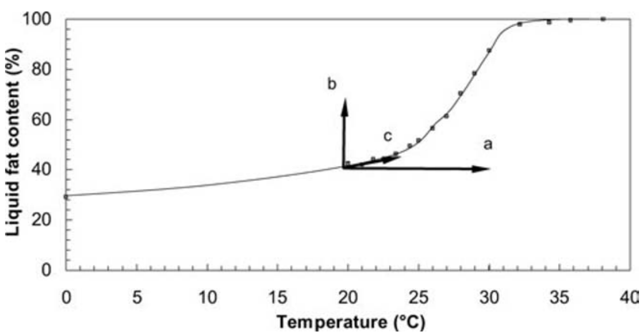

Figure 5. Liquid fat content as a function of temperature with three possible process paths for extrusion processing of semi-solid chocolate.

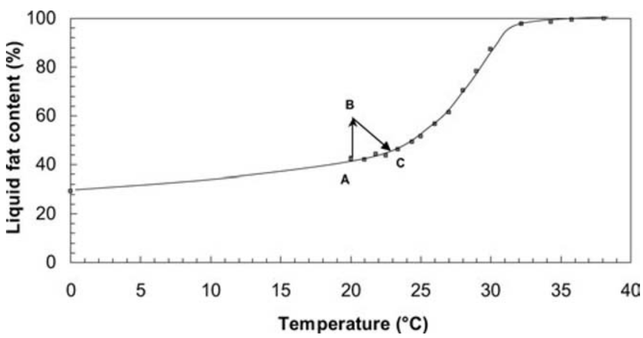

Figure 6. Liquid fat content as a function of temperature with suggested process path for extrusion processing of semi-solid chocolate.

increase of the liquid fat content during the extrusion (A to B) and the subsequent return to the equilibrium curve (B to C). After recrystallization it can be anticipated that the extrudate will subsequently return to the ambient temperature given by A.

FINITE ELEMENT METHOD

To calculate the spatial distribution of energy, stress and rate of deformation from the governing equations resulting from the model outlined above, we have used Forge2® (Transvalor S.A., Sophia Antipolis, France) a commercially available finite element software designed for solving two-dimensional elastic-plastic and rigid-plastic deformation problems. The method is Lagrangian, incremental and uses an implicit solution algorithm (for a discussion of different numerical approaches to plastic flow problems, see Wagoner and Chenot, 2001). To cope with the problem of mesh distortion occurring in large-strain deformations such as extrusion, the software uses an automatic mesh generator which maps the current solution onto a new mesh whenever required. Previous work by Mulji and Mackley (2003, 2004) has shown that analytic and numerical calculations for perfectly plastic materials are in good agreement with experimental pressure drop measurements for plastic deformation within convergent dies.

The version of Forge2® we used did not allow the definition of user-defined state variables. Changes of the liquid fat content ϕ and their effect on mechanical properties were therefore implemented via the *temperature* variable used by the software by using a 'pseudo-temperature' corresponding to the liquid fat content, even though the process was considered to be isothermal.

RESULTS

Change of Liquid Fat Content During Extrusion

We first calculate the *average* change of liquid fat content during an extrusion, as this is easier to compare with our existing experimental data (Engmann and Mackley, 2005), but does not contain information about the spatial distribution of the phase contents. Subsequently, we calculate the *distribution* of the liquid fat content numerically, using Forge2. The two calculation procedures are shown schematically in Figure 7.

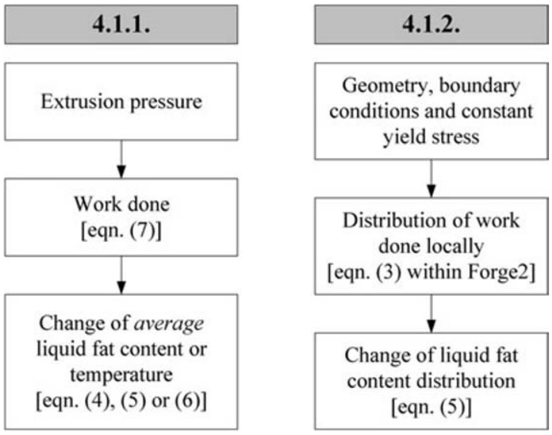

Figure 7. Calculation procedures for change in liquid fat content (average and spatial distribution).

Average change of liquid fat content

To calculate the average change of temperature and liquid fat content in the material during extrusion processing, a macroscopic, or global energy balance (see, e.g., Bird *et al.*, 2002) around the extrusion die can be made, giving (under the assumption that the system behaves adiabatically)

$$\frac{w}{\rho} = e_2 - e_1 = \frac{P}{\rho} - \frac{1}{2}U\left[\left(\frac{D_0}{D}\right)^4 - 1\right] \quad (7)$$

where (w/ρ) is the work done per unit mass $(e_2 - e_1)$, the change of the average internal energy per unit mass, P the pressure difference across the extrusion die and ρ the density of the material (which is assumed to be incompressible). The second term on the RHS of equation (7), which reflects the change in kinetic energy, can be neglected for our purposes, since it is much smaller than $\Delta P/\rho$ for extrusion dies with common diameter ratios.

Using experimental pressure drop data for extrusion at 20°C and caloric data given in Table 1 (interpolating between the solid and liquid heat capacities in the semi-solid range), Table 2 gives predicted changes for each of the model case scenarios, namely (a) heating only, (b) phase change only, and (c) a mixed regime. Temperature increases are predicted using assumptions (a) or (c) and this is in conflict with the experimental observation of no temperature increase *immediately after extrusion*. We therefore conclude that during extrusion, all the work done goes into a phase transformation of crystal triglycerides to the liquid state. The gradual temperature rises experimentally observed at *longer times after extrusion processing*, however, does compare reasonably well to those predicted by assumption (c), indicating that in this stage of the process some latent heat of recrystallization is eventually converted to thermal heat, as schematically shown in Figure 6(b) and (c).

Distribution of liquid fat content

Using the Forge2 finite element software (with a yield stress value of $\tau_y = 0.866$ MPa) it was possible to compute the stress distribution within the convergent section of the

Table 1. Caloric properties of chocolate. Data from Chevalley *et al.* (1970)

	Temperature (°C)	Dark chocolate	Milk chocolate	Cocoa butter
Heat capacity, solid ($kJ\ kg^{-1}\ K^{-1}$)	0	1.30	1.55	2.22
	10	1.34	1.68	2.38
Heat capacity, liquid ($kJ\ kg^{-1}\ K^{-1}$)	40	1.38	1.55	2.01
Enthalpy of fusion ($kJ\ kg^{-1}$)	10–40	46.5	44.4	157

Table 2. Change in ϕ and T for different chocolates and extrusion pressures, for a temperature of $T = 20°C$ of the initial material. Extrusion pressures from previous work (Crook, 1997; Ovaici, 1999; Mulji, 2000), temperature rise data from Mulji (2000).

					Heat capacity only (a)	Latent heat only (b)	Heat capacity and latent heat (c)		Temperature rise after extrusion processing (experimental data from Mulji, 2000)
	w_f	$d\phi$ (K^{-1})	P (MPa)	w/ρ (kJ kg^{-1})	ΔT (K)	$\Delta\phi$	ΔT (K)	$\Delta\phi$	ΔT (K)
Milk chocolate	0.28	0.02	4.5	3.46	2.2	0.08	1.4	0.03	—
			5	3.85	2.4	0.09	1.6	0.03	1.4
			5.5	4.23	2.6	0.09	1.7	0.04	—
Dark chocolate	0.29	0.04	12.5	9.62	6.0	0.22	2.8	0.12	—
			14	10.77	6.7	0.23	3.1	0.14	2.8
			15.5	11.92	7.5	0.26	3.5	0.15	—
Cocoa butter	1.00	0.01	4.5	4.62	2.3	0.03	1.3	0.01	—
			5	5.13	2.5	0.03	1.4	0.01	1.6
			5.5	5.64	2.8	0.04	1.6	0.02	—

die. Forge2 is able to calculate the local energy dissipation at any spatial position and normally this would be converted to a change of the local temperature. In our case however, we have re-expressed this as a local change in liquid fat phase content according to equation (5), and Figure 8 shows the distribution of liquid fat content in the plane of symmetry (r, z) of a frictionless axisymmetric extrusion die. From the figure it can be seen that the liquid fat content increases near the die entry and near the capillary wall.

Figure 9 shows the radial distribution of the liquid fat content at the exit plane of three extrusion dies with different area reductions $s \equiv (D_0/D)$. With increasing area reduction, the local work done and liquid fat content becomes greater everywhere on the exit plane and the profiles appear to become sharper towards the die exit corner, i.e., the extrudate surface.

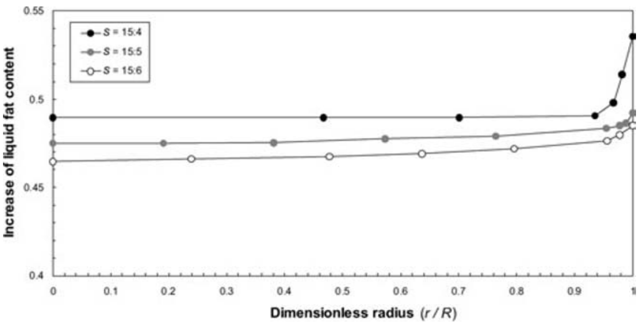

Figure 9. Change of the liquid fat content from the initial value to its value in the exit plane of axisymmetric orifice dies as a function of radial position. Plotted for three extrusion dies with different area reductions $s = D_0/D$.

Effect of Increasing Liquid Fat Content on Extrusion Pressure

We now consider the effect of the change in liquid fat content on the extrusion pressure, first globally (considering extrusion pressure and average liquid fat content) and then locally (considering changes in yield stress and liquid fat content distribution). The calculation procedures are shown schematically in Figure 10.

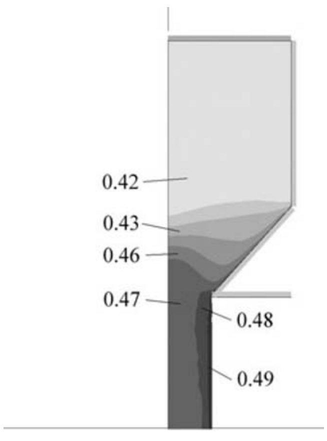

Figure 8. Spatial distribution of liquid fat content in chocolate during an axisymmetric extrusion ($D_0/D = 15:5$, $L = 0$, $\alpha = 45°$).

Effect on extrusion pressure during a sequence of extrusions

In the companion paper to this one (Engmann and Mackley, 2005), we showed that there was a progressive reduction of the extrusion pressure for successive multipass extrusions with no time delay between extrusions as shown for example in Figure 8 of that paper. We now estimate how the change of the liquid fat content affects the extrusion pressure in a sequence of extrusions. This is done by calculating the change of the average liquid fat content for each extrusion, using equations (6) and (7) and calculating the pressure for each extrusion 'pass' from the data shown in Figure 4 for a milk chocolate. These data can be fitted by the empirical equation

$$\frac{P}{\text{MPa}} = \frac{1}{1 - (1 - \phi/0.69)} - 1.5 \qquad (8)$$

Using equations (6)–(8) for three different initial conditions of the liquid fat content (0.52, 0.58 and 0.61),

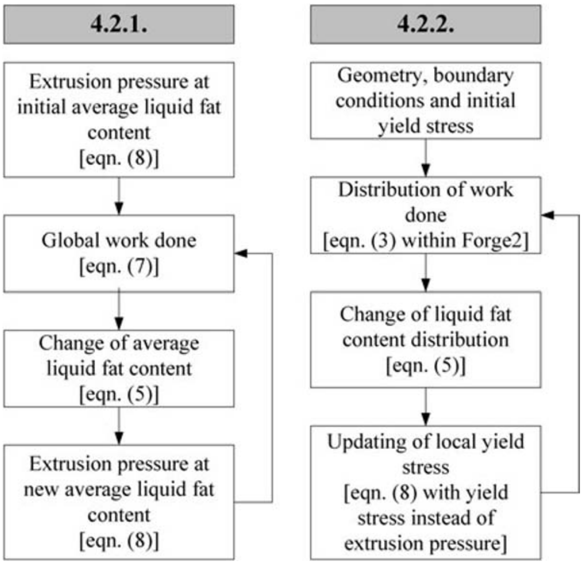

Figure 10. Calculation procedures for change in extrusion pressure as an effect of the change in liquid fat content (for a sequence, respectively a single extrusion).

the evolution of extrusion pressure and liquid fat content is calculated and shown in Figure 11. As the liquid fat content grows, the extrusion pressure decreases and so does, by virtue of equation (7), the amount of work done during the next extrusion. The curves for the extrusion pressure and liquid fat content therefore progressively flatten towards an asymptotic value. Experimental data for the reduction in extrusion pressure are also shown in the figure (large filled circles), which show qualitatively similar behaviour although with a lower relative reduction in extrusion pressure.

Effect on extrusion pressure in a single extrusion

The calculation in the preceding section assumed that the change of the yield stress during a single extrusion does not significantly change the flow and extrusion pressure compared with an identical extrusion process of a constant yield stress material, although it does affect subsequent processing, e.g., in a sequence of extrusions. The soundness of this assumption is now investigated by finite element calculations.

Finite element analyses were conducted as described earlier, but the (local) yield stress was now made dependent on the local liquid fat content, calculated on the basis of equations (3) and (5). A proportional relationship between extrusion pressure and yield stress was assumed, and the pressure data shown in Figure 4, fitted by equation (8) were thus used to predict the dependence of yield stress on liquid fat content. The results for a number of orifice dies are shown in Figure 12. The extrusion pressure, which has been normalized by the *initial* yield stress τ_y^0, has been plotted against a dimensionless variable reflecting the dependence of the yield stress on liquid fat content, $\Delta\tau_y/\Delta\phi$, relative to the energy per unit volume required to cause a complete phase change from solid to liquid, $\rho w_f H_m$. The values for this dimensionless variable used in the calculation span the range of physically realistic values for chocolate and cocoa butter.

These results suggest that the influence of the work softening on the extrusion pressure for a single extrusion is quite weak, at least for dies with moderate diameter reductions. This is in contrast to the substantial influence that the work softening characteristics had during a sequence of extrusions as discussed previously and seen experimentally.

At this stage we are not entirely certain why the numerical model predicts such a small effect on a single extrusion pressure compared to the pressure reduction in an extrusion sequence that was measured and predicted earlier. A possible explanation for this discrepancy could be the link we have used between the yield stress and the liquid fat content, which is based on the assumption that this relation is essentially the same as that between the *single extrusion pressure* and *initial liquid fat content*, shown in Figure 4 and expressed by equation (8). It is possible that the extrusion process has a stronger effect on the local yield stress than that presented by Figure 4. In addition, it can be seen from Figure 8 that the predicted distribution of fat phase modification is highly non-uniform, which has also been experimentally observed

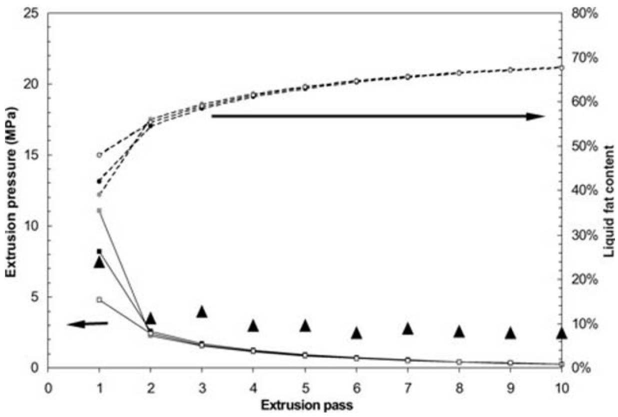

Figure 11. Sequence of extrusion pressures and corresponding liquid fat contents at the beginning of each extrusion pass. Circles/dashed lines correspond to three different evolutions of liquid fat (depending on the starting value), squares/solid lines to the evolution of extrusion pressure for the same starting values of liquid fat content and triangles to an experimentally observed extrusion pressure sequence.

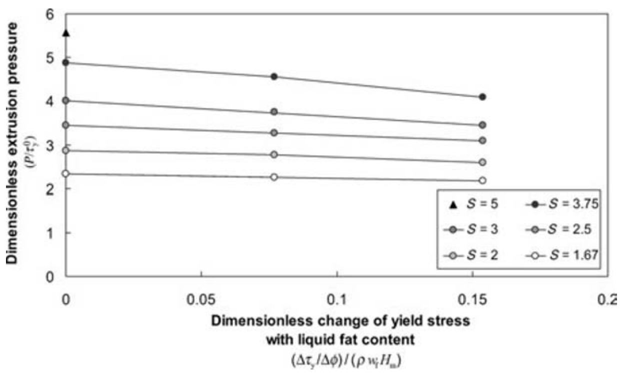

Figure 12. Extrusion pressure as a function of yield stress dependence on liquid fat content for different diameter reductions $s = D_0/D$.

and reported in a recent study of chocolate cold extrusion (Mulji *et al.*, 2003). During each extrusion, material is locally subjected to the highest amount of work near the corners of the extrusion die and consequently on successive extrusions the fat phase distribution may become even more non-uniform and thereby influence the overall extrusion pressure.

CONCLUSIONS

This paper has shown how extrusion processes can be modelled using the concepts of perfect plastic flow together with a microstructural variable that controls the yield stress, which is linked to the amount of work dissipated. The model has been developed for the extrusion of semi-solid chocolate and cocoa butter through axisymmetric dies and has explained some of the phenomena observed during the extrusion of these materials.

The results demonstrate the importance of considering microstructural changes during extrusion, both for predicting product properties and for understanding the extrusion process itself. While the model has been specifically developed to describe extrusion of semi-solid chocolate, the approach could also have applications to other extrusion and forming processes.

If caloric data and phase diagrams of higher accuracy than presently available could be determined, this would allow a further comparison of the model predictions with the experimental observations. Further, the experimentally observed re-hardening of extruded chocolate extrudate, associated with a re-crystallization of the fat matrix has not yet been incorporated and provides a next logical extension to the model.

NOMENCLATURE

c	specific heat capacity
D	die exit diameter
D_0	die entry diameter
$\mathbf{D}$	rate-of-deformation tensor
e	internal energy
H_m	enthalpy of melting
L	die land length
p	isotropic stress (pressure)
P	extrusion pressure
Q	volumetric flow rate
s	reduction ratio
T	temperature
U	ram velocity
$\dot{w}$	rate of work done
w	work done
w_f	fat content

Greek symbols

α	die entry angle
ϕ	liquid fat content
ρ	mass density
σ	Cauchy stress tensor
τ	deviatoric stress tensor
τ_y	yield stress

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